



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 187e

Sodium Tetraborate Decahydrate (Borax)

pH Standard

This Standard Reference Material (SRM) is intended for use in preparing solutions for calibrating electrodes for pH measuring systems. SRM 187e, Sodium tetraborate decahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{ H}_2\text{O}$) was prepared to ensure high purity and uniformity. However, this SRM is certified **ONLY** as a pH standard, pH(S), not as a pure substance. A unit of SRM 187e consists of 30 g of sodium tetraborate decahydrate.

Certified Values and Uncertainties: The certified pH(S) values provided in Table 1 correspond to $\log(1/a_{\text{H}})$, where a_{H} is the conventional activity of the hydrogen (hydronium) ion referred to the standard state ($p^\circ = 1 \text{ atm} = 1.01325 \times 10^5 \text{ Pa}$) on the scale of molality. The values were derived from emf measurements of cells without liquid junction by the primary measurement method [1,2]. **NOTE:** These certified values apply **ONLY** to SRM 187e. Minor variations of pH(S) values (of the order of a few thousandths of a unit) may be expected to occur between SRM lots.

The uncertainty in the certified value, U , is calculated as $U = ku_c(y)$, where $u_c(y)$ is the “combined standard uncertainty” calculated according to the ISO/NIST Guides [3]. The value of $u_c(y)$ is intended to represent the combined effect of the following uncertainty components associated with the primary measurement method and material homogeneity: extrapolation to obtain the acidity function, $p(a_{\text{H}}\gamma_{\text{Cl}})^\circ$; standard electrode potentials, E° ; material homogeneity; molality of HCl, b_{HCl} , used for determining E° ; measured cell potentials; correction to the standard pressure for H_2 gas; mean activity coefficient of HCl at b_{HCl} ; gas constant; temperature; Faraday constant; the molality of NaCl; and the uncertainty [4,5] of the conventional calculation of $\log \gamma_{\text{Cl}}$ (Bates-Guggenheim convention [6]). Current expert opinion [4,5] has assessed the uncertainty attributable to the Bates-Guggenheim convention as 0.010 pH (95 % confidence interval). The value of $u_c(y)$ has been multiplied by a coverage factor, k , obtained by the Student's t -distribution for effective degrees of freedom at the given temperature and a 95 % confidence level. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST [7]. The certified pH(S) values and their expanded uncertainties, U , are stated in Table 1.

Expiration of Certification: The certification of this SRM is valid until **31 March 2009**, within the measurement uncertainties specified, provided the SRM is handled in accordance with instructions given in this certificate (see “Instructions for Use”). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

The experimental work leading to the certification of this material was performed by K.W. Pratt of the NIST Analytical Chemistry Division.

Statistical consultation was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

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A solution of molality 0.01 mol/kg is recommended for the calibration of pH measuring systems. The pH(S) and the expanded uncertainty, U , of this solution as a function of temperature, t , are given in Table 1.

Table 1. Certified pH(S) Values and Expanded Uncertainties (95 % confidence)

$t/^{\circ}\text{C}$	pH(S)	$u_c(y)^a$	k_{cov}	U
5	9.393	0.0053	1.96	0.010
10	9.333	0.0053	1.96	0.010
15	9.278	0.0053	1.96	0.010
20	9.230	0.0053	1.96	0.010
25	9.186	0.0052	1.96	0.010
30	9.146	0.0052	1.96	0.010
35	9.110	0.0052	1.96	0.010
37	9.096	0.0052	1.96	0.010
40	9.077	0.0052	1.96	0.010
45	9.047	0.0052	1.96	0.010
50	9.020	0.0052	1.96	0.010

^a $u_c(y)$ is the combined standard uncertainty, which includes u_c for the measurement (see Table 2, below) and the standard uncertainty of the Bates-Guggenheim Convention (0.0050) [4,5].

Reference Values: To attain traceability to the NIST reference pH(S) values for SRM 187e when traceability to the SI is not necessary, the uncertainty of the Bates-Guggenheim convention is excluded from the uncertainty calculation. The respective pH(S) values in Table 2 are identical to those in Table 1 but are listed to the number of decimal places corresponding to 2 significant figures for the corresponding expanded uncertainty, U_R :

$$U_R = k_R u_c(\text{measurement})$$

where k_R is the coverage factor for U_R . NIST Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty [7].

Table 2. Reference pH(S) Values and Expanded Reference Uncertainties (95 % confidence)

$t/^{\circ}\text{C}$	pH(S)	$u_c(\text{measurement})^a$	k_R	U_R^a
5	9.3934	0.0017	2.1	0.0034
10	9.3328	0.0016	2.1	0.0033
15	9.2784	0.0016	2.1	0.0033
20	9.2295	0.0016	2.1	0.0033
25	9.1855	0.0016	2.1	0.0033
30	9.1459	0.0015	2.1	0.0031
35	9.1099	0.0015	2.1	0.0031
37	9.0965	0.0015	2.1	0.0031
40	9.0772	0.0015	2.1	0.0031
45	9.0472	0.0015	2.1	0.0031
50	9.0195	0.0016	2.1	0.0032

^a $u_c(\text{measurement})$ and U_R each include all components associated with the measurement method and assessment of material homogeneity, but do not include the uncertainty of the Bates-Guggenheim Convention (0.0050) [4,5].

NOTICE AND WARNINGS TO USERS

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Source of Material: The sodium tetraborate decahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{H}_2\text{O}$) was obtained from a commercial company. This material conforms to the specifications of the American Chemical Society for primary standard chemicals [8].

Storage: SRM 187e is stable when stored in its original container, with the cap tightly closed, in a dry environment, and under normal laboratory temperatures.

INSTRUCTIONS FOR USE

Drying Instructions: Use as received. The salt must **NOT** be dried in an oven. Do not store in a desiccator before use.

Preparation of Carbon Dioxide-Free Water: Carbon dioxide free water must be used for making the solutions. This water must be prepared either by (1) boiling a good grade of distilled water (conductivity $< 2 \mu\text{S}/\text{cm}$) for 10 min and guarding it with a soda-lime tube while cooling or (2) dispensing water directly from a deionization-based point-of-use system into the vessel used to prepare the buffer solutions (resistivity $> 17 \text{ M}\Omega\cdot\text{cm}$, conductivity $< 0.06 \mu\text{S}/\text{cm}$).

Preparation of the 0.01 mol/kg Solution: Quantities denoted by m_{W} and associated numerical factors in this paragraph include the effect of air buoyancy, i.e., they correspond to the balance indication in units of mass obtained in the laboratory (the “balance reading”). Weigh by difference approximately 3.7 g of SRM 187e, $m_{\text{W},187\text{e}}$, to an accuracy of 1 mg, into a clean, dry, 1-liter polyethylene bottle. Add a quantity of CO_2 -free water, equal to 261.841 multiplied by $m_{\text{W},187\text{e}}$, to an accuracy of 0.1 g. Shake until the solid has totally dissolved. Gravimetric preparation in this manner eliminates the need to weigh exactly predetermined masses of solid samples. Proportionately smaller quantities of SRM 187e may be used in this preparation, provided that $m_{\text{W},187\text{e}}$ exceeds 1.0 g.

The corresponding apparent mass in air yielding a molality equal to $0.010\,000 \text{ mol}\cdot\text{kg}^{-1}$ is 3.8191 g $\text{Na}_2\text{B}_4\text{O}_7\cdot 10\text{H}_2\text{O}$ per 1000.0 g H_2O . As an alternative to the above procedure, solid SRM 187e may be added in this proportion to CO_2 -free water, yielding an identical solution to that obtained above. However, any increased uncertainty associated with missing the target value of the SRM 187e or the water must be taken into account.

Stability of Prepared Solution: Solutions should be discarded after one month or sooner if sediment appears, or if it has been exposed repeatedly to air containing carbon dioxide. To avoid contamination of the buffer solution with atmospheric carbon dioxide, keep the bottle capped except when removing a portion of the solution.

REFERENCES

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- [3] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st ed.; ISO: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at <http://physics.nist.gov/Pubs/>.
- [4] Buck, R.P.; et. al.; *Measurement of pH. Definition, Standards, and Procedures*, IUPAC Recommendation 2002, Pure Appl. Chem, Vol. 74, No. 11, p. 2169 (2002); also available on-line at <http://www.iupac.org/publications/pac/2002/pdf/7411x2169.pdf>.
- [5] Baucke, F.G.K.; Anal. Bioanal. Chem., Vol. 734, p. 772 (2002).
- [6] Bates, R.G.; Guggenheim, E.A.; *A Report on the Standardization of pH and Related Terminology*; Pure Appl. Chem., Vol. 1, p. 163 (1960).
- [7] May, W.; et. al.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000).
- [8] *Reagent Chemicals*; 8th ed.; American Chemical Society: Washington, DC (1993).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.